

## The Design and Operation of Fluidized-Catalyst Phthalic Anhydride Plants.

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The catalytic oxidation of naphthalene to phthalic anhydride by atmospheric oxygen is a reaction admirably suited for a fluidized reactor. Early attempts to produce phthalic anhydride on a commercial scale by this means ran into serious difficulties. This was because of mistakes in design and a lack of appreciation of many of the factors involved. It is proposed in this paper to describe how these difficulties have been overcome and also to indicate the essential features in design and operation for the trouble-free manufacture of phthalic anhydride by this method.

### Fluidized-bed v. Fixed-bed.

Phthalic anhydride has been manufactured by the oxidation of naphthalene in fixed-bed reactors for more than forty years. During this time considerable design and operational experience has been accumulated. Nevertheless, it is widely known that such reactors have inherent defects which make impossible the desired control of reaction conditions. The advent of the fluidized-bed reactor has made possible not only a much closer approach to these ideal reaction conditions but also several other important advantages which favourably influence the economics of the process. Fig. 1. is a flow diagram of a fluidized-catalyst phthalic anhydride plant and below are listed some of the important advantages gained by this method of manufacture.

- (a) Greater Safety. The presence of finely divided catalyst in the reactor prevents explosions and so permits operation well inside the explosive range of air:naphthalene ratios.
- (b) More precise control of reaction conditions. The high thermal conductivity and good heat-exchange characteristics of this fluidized-catalyst bed permits an exact control of reaction temperature without the formation of hot spots. Temperatures varying only by one or two °F. can be held throughout a fluidized bed containing up to 30 tons of catalyst or more. Pressure, temperature, air:naphthalene ratio and contact time can each be varied, independently and quickly, so permitting a rapid and exact adjustment of operating conditions to meet particular requirements.
- (c) More efficient condensation of phthalic anhydride. A substantially lower air:naphthalene ratio makes possible the more efficient condensation of the reaction product because of its greater partial pressure in the product gases. Operating the reactor at increased pressure permits the condensation of 50% or more of the product directly as liquid, the balance being condensed as solid in switch condensers. The lower concentration of oxygen in the product gases, compared with that obtaining in fixed-bed plants, reduces the hazard of fire and explosion down stream from the reactor.

- (d) Liquid naphthalene feed. Fixed-bed phthalic anhydride reactors are fed with a mixture of air and naphthalene vapour involving the use of a separate evaporator. This can give trouble. With a fluidized-catalyst reactor liquid naphthalene is pumped directly into the reactor.
- (e) Catalyst charging. The tedious operation of charging catalyst pellets into the tubes of the fixed bed reactor is avoided. On the fluidized-bed plant catalyst can be moved pneumatically from the reactor to the catalyst storage vessel or in the opposite direction, even whilst the plant is operating.
- (f) Operation at increased pressure. The operation of fluidized-catalyst reactors at increased pressure (1 to 2 atm. gauge) permits a substantial increase in the output of a particular unit compared with its capacity at atmospheric pressure. Air compression cost must be balanced against higher throughput in order to determine the optimum operating pressure. The removal of the additional heat liberated as throughput is increased presents no serious design problems.
- (g) High purity product. The precise control of reaction conditions in a fluidized catalyst reactor results in the formation of a crude product of high purity. The redistillation process is thereby considerably simplified, and a final sales product of a high standard of purity is obtained. Table 1. gives the results of the analyses of a number of commercial samples of phthalic anhydride all taken independently and analysed blind. The higher quality of the samples from the fluidized-catalyst plants is apparent.

R. Landau and H. Harper (7) have given a critical, historical review of the development of phthalic anhydride technology. They mention the difficulties that have troubled the pioneers of the fluidized catalyst process. How these difficulties have been overcome is described below.

### Condensation

The early fixed-bed phthalic anhydride plants were small and operated with high air:naphthalene ratios. The product was condensed in the so-called barns or hay boxes, which were large vessels, usually several in series. They served to slow down the gas velocity and wire net in the boxes provided additional surface for the condensation of the needle-shaped crystals of phthalic anhydride. The natural cooling of the boxes was usually sufficient for efficient condensation. With large production units, however, such barns are impracticable on the grounds of cost, danger and difficulties in handling the solid product. In modern plants they have been replaced by tubular heat exchangers operated as switch condensers. During condensation the tubes are cooled by water or oil at 35 to 40°C. Lower temperatures would promote the condensation of excessive amounts of water vapour with the concomitant formation of phthalic and maleic acids. For melting out, the cold water is replaced by high pressure steam and the cold oil by hot oil. A temperature high enough to dehydrate phthalic acid is essential otherwise it will accumulate inside the condenser reducing its efficiency and capacity and finally causing blockages.

Different types of tubular switch condensers have been described (3, 12); some are too complicated to be practicable in large production units. Besides simple heat-exchange considerations, other factors are involved in the design of efficient switch condensers. e.g., (1) Spare capacity is necessary to permit periodic cleaning of the tube bundles without interrupting production. (2) The capacity of the individual condensers must be such that the switch operation is relatively infrequent, otherwise there would be an excessive thermal load due to the relatively high heat capacity of the condensers which could be even more than the heat required to melt the phthalic anhydride. (3) The construction of the condensers should be such that

TABLE 1.

Source of Sample	Phthalic Anhydride %	Phthalic Acid %	Maleic Anhydride %	Naptha-quinone %	Iron p.p.m.	Ash %	Crystallising Point °C	Colour Hazen Units	Colour after 2 hours at 250°C Hazen. Units
Fixed Bed A.	99.4	0.17	0.02	not detected	1.7	0.0006	130.6	45	113
Fixed Bed C.	99.1	0.65	0.17	"	2.0	0.0056	130.3	100	250
Fixed Bed E.	99.3	0.59	0.04	"	0.4	0.0010	130.3	90	225
Fluid Bed B.	99.6	0.08	0.01	"	0.4	0.0004	130.9	30	77
Fluid Bed D.	99.7	0.15	0.02	"	1.0	0.0028	130.8	65	97
Fluid Bed F.	99.3	0.40	0.01	"	1.9	0.0002	130.5	30	70
Fluid Bed G.	99.5	0.40	0.08	"	0.7	0.0004	130.7	25	60

the heat capacity is kept to a minimum. The oil system for cooling and melting is therefore preferred rather than the water/high pressure steam system. (4) The design should be such as to minimize or eliminate fire and explosion hazards. This can be achieved by the use of a closely packed gilled tube bundle. (5) It is not sufficient in condensers of this type merely to provide adequate cooling surface. Provision must also be made for ensuring that the product gases have the opportunity of contacting preformed phthalic anhydride crystal surfaces, otherwise even with more than adequate cooling, solid deposition will not necessarily occur. Fig. 2. shows a 6 ft. length of finned tube enclosed in glass pipe-line. The tube was cooled by passing water through it. Air saturated with phthalic anhydride vapour at  $140^{\circ}\text{C}$  was passed downwards through the annulus between the finned and the glass tubes. Solid phthalic anhydride was deposited on preformed crystals rather than on the cooler base portions of the finned tube. Vapour super-saturated with phthalic anhydride will leave the tube if the speed of the gas exceeds a low minimum value.

Fig. 3. shows diagrammatically a switch condenser which meets these requirements (14, 15). It consists of a relatively light gauge steel box packed with rows of vertical gilled tubes connected to manifolds. During the condensation part of the cycle, cold oil ( $40^{\circ}\text{C}$ ) is passed through these tubes. The product gases from the fluidized reactor enter the condenser and at first pass at relatively low velocity, more or less straight through the banks of gilled tubes. Massive, solid phthalic anhydride condenses on the first banks which become blocked and an increasing amount of the product gas passes through the gaps at the ends of the banks of gilled tubes. These gaps at opposite ends of adjacent rows prevent excessive build up of back pressure before a substantial quantity of phthalic anhydride is condensed. The increased gas velocity as the gas flows parallel to the rows improves heat exchange and gives good contacting of the product vapours with preformed phthalic anhydride crystals. As the product gases progress through the condenser the concentration of phthalic anhydride decreases and condensation occurs on the later banks of gilled tubes in the form of long needles. The close packing of the tubes no doubt also has a filtering action on any gas borne particles of phthalic anhydride.

At a predetermined time a condenser is taken off stream and the cold oil replaced by hot. The phthalic anhydride melts and is run to storage. The hot oil is then switched back to cold; after a period for cooling the empty condenser is again put on stream. It is essential to use a thermally stable oil for this purpose.

The United Coke and Chemical Company's plant operates on eight of these switch condensers together with a ninth stand-by for periodic special cleaning. At normal throughput a sequence of six condensing and two melting is employed. The capacity of the boxes is such that switching is carried out at long intervals and little or no advantage would be gained from automatic operation. Analyses have shown that a small amount of slip takes place at the commencement of the condensation i.e., when the tube surfaces are clean and cooling must be at its best, but when there is little or no solid phthalic anhydride to promote condensation. Long intervals between switching reduces the loss by slip to a very low value.

These condensers have been tested for long periods with air:naphthalene ratios as high as 30:1 and the results obtained indicate that they could be used advantageously with fixed-bed reactors.

Fluidized-catalyst reactors, operating with low air:naphthalene ratios and at increased pressure can advantageously condense an appreciable proportion of the product directly as liquid (2) and so decrease the thermal load on the switch condensers.

## Filtration

In order to prevent contamination of the product and loss of an expensive catalyst it is essential in a fluidized-bed phthalic anhydride plant efficiently to filter the product gases. Early design proposals (4) suggested the use of cyclones. In the first production unit, the Sherwin Williams Company (6) developed the use of porous ceramic filters for the complete elimination of the catalyst dust at high temperature. Porous stainless steel filters have been used in pilot plants. The United Coke and Chemicals Company (13) have developed a fibre-glass filter. It consists essentially of a perforated steel cylinder (Fig. 4) having a spigot at each end. The upper spigot is threaded and serves to connect the filter to a collector box. The lower spigot is closed and serves to protect the filter. It also acts as a journal whilst the filter is being wrapped. The wrapping, which is done on a simple machine, consists of alternating layers of continuous-filament glass cloth and super-fine fibre-glass mat. The first 5 layers (3 cloth and 2 mat) are held in position by an open wound helix of glass tape. Another similar series of cloth, super-fine mat, cloth and tape, is added followed by an overlapping spiral wrap of glass tape and the whole bound firmly but not too tightly with jubilee or similar metal clips at 18" intervals. The overlapping ends of the glass cloth are secured in grooves at the two ends of the filter tube, by means of fibre-glass cord. This is an additional precaution to prevent end leakage. Four separate collector boxes are used, each carrying several of these fibre-glass filters. By means of a process controller each bank of filters can be blown back in turn to detach filter cake from the outer surface. More than ten years of operational experience has shown that these filters are a reliable means of obtaining 100% filtration of catalyst dust from the product gases at a temperature of about 300°C. They have advantages over porous ceramic filters in that they are not brittle, they can be quickly and relatively cheaply re-wrapped and if necessary, can be made to operate at low back pressure. On blow-back, the slight "give" in the fibre-glass wrapping tends to cause better removal of the accumulated filter cake. The back pressure across the filters depends, of course, on the surface area of the filter and the volume of gas passing. It is customary to operate fluidized-catalyst phthalic anhydride plants under pressure so a filter pressure drop of 4 to 6 lb. p.s.i. or more can be accommodated. Filter candles up to 12 ft. in length have been successfully used. Experiments have also been carried out in which these filters have been operated at 10 to 20 ins. w.g. back pressure for prolonged periods at 300°C.

The positioning of the filtration unit in the plant is important and is discussed below.

### The Fluidized-bed Catalyst.

The physical and chemical nature of the catalyst is of great importance for the trouble-free operation of a large fluidized-bed phthalic anhydride plant. Not only must it have high and specific reactivity but also good fluidizing properties, high attrition resistance and long life. The catalysts used in the early days of this new technique were formulated on the grounds of their catalytic activity with little or no attention to their fluidizing properties and attrition resistance. It is possible in laboratory apparatus and small pilot plants to obtain reasonably good fluidization and excellent conversions of naphthalene into phthalic anhydride with a wide range of catalysts. Nevertheless, in full scale reactors troubles can arise from the use of friable catalysts with the consequent rapid accumulation of an excessive proportion of fines in the fluidized bed. This may bring about uneven fluidization and an undesirably high concentration of fines in the expanded phase. Both these states can give rise to trouble; the latter is probably a contributory cause to "after burning" (see below), whilst the former may bring about the formation of static pockets of catalyst. Such pockets in contact with the reactor walls can act as naphthalene condensers. The mixture of solid naphthalene and catalyst so

formed, if suddenly disturbed and ignited can react very vigorously with the production of excessively high temperatures within the fluidized bed.

Fluidized-bed phthalic anhydride catalysts must have a fairly high specific surface and a relatively large pore diameter. These properties are not compatible with high attrition resistance so it is necessary to compromise. It has been found possible to formulate a highly specific catalyst with the required attrition resistance and long life. In a favourably designed reactor, size degradation is surprisingly slow and present indications are that it should be possible to maintain the activity and good fluidizing properties of the catalyst with a replacement level as low as 10% per annum.

#### After Burning

One of the most serious and dangerous difficulties which beset pioneers in this field was "after burning", i.e. the ignition of the product vapours above the fluidized-catalyst bed with a consequent excessive and sometimes disastrous rise in temperature. This has already been described in some detail (1). In the absence of effective temperature control in the expanded phase, temperatures as high as 750 to 1000°C or more may be reached with consequent serious damage to the filters, catalyst and reactor. The most likely explanation of this phenomenon is excessive slip of naphthalene through the fluidized catalyst dense phase occasioned by poor fluidization, particularly slugging, spouting, rat-holing or an excessive proportion of bubble phase in the boiling bed. A small amount of particularly active, static catalyst lodged somewhere above the fluidized-bed possibly starts the exothermic oxidation of this naphthalene. (11) and with a relatively rapid rise of temperature initiates the after burning. Whatever the cause, after burning can be prevented by keeping the temperature of the expanded phase well below that of the bed. Various methods of doing this can be used:-

- (1) Cooling tubes in the expanded phase. Although the heat-exchange coefficient on the gas side of the tubes is not favourable, the dust concentration in the gas increases the amount of cooling over that which would obtain with a clean gas.
- (2) Quench Zone. A specially cooled extension of the fluidized bed to cool the gases leaving the reaction zone has been described by the American Cyanamid Company (1). It necessitates the use of an additional distributor plate, special cooling coils and a standpipe. This upper part of the fluidized bed is termed the "quench zone" and it is recommended that the temperature of this zone is kept at least 50°C below that of the main fluidized bed. Such an arrangement could, if not properly designed and operated, lead to an increased rate of catalyst attrition (see below).
- (3) Quench Gas. The temperature above the bed can be effectively controlled by the introduction of cold air, or better, cold inert gas. This simple and practicable method has the advantage of reducing the concentration of phthalic anhydride in the product gases, which, of course, is not good from the condensation angle.
- (4) Recycling cooled catalyst. Recycling cooled catalyst through the expanded phase can be used to cool the gases leaving the boiling bed. This, however, may result in increasing catalyst attrition to an objectionable rate.
- (5) External cooling. If the catalyst filters are housed in separate vessels, then it is possible to cool the product gases by utilizing the surface of the interconnecting pipe-lines. This is effective in preventing after burning but involves catalyst circulation at high velocity, which may contribute to catalyst attrition (see below).

Another factor which makes an important contribution to minimising the risk of after burning is low oxygen partial pressure in the product gases. This is achieved by operating at the lowest possible air:naphthalene ratio compatible with crude product purity and bed temperature.

Trouble from after burning has been virtually eliminated from modern fluidized-catalyst phthalic anhydride plants by the use of one or more of the above devices.

#### Reactor Design and Catalyst Attrition

There is an upper limit to the attrition resistance which can be built into an acceptable naphthalene oxidation catalyst. Reactor design should therefore be such as to keep catalyst attrition to a minimum; otherwise excessive catalyst replacement will be necessary in order to prevent a built up of an excessive proportion of catalyst fines. This also places a limit on the fluidizing gas velocity and emphasises the important advantage of operating fluidized-catalyst reactors at increased pressure in order to obtain the necessary contact time and economic throughput. Three design features are involved in minimising catalyst attrition viz., (1) the disposition of the catalyst cooler, (2) the disposition of the filters and (3) the distributor plate. These are discussed below.

- (1) The Catalyst Cooler. Some of the earlier patents (16) describe fluidized catalyst reactors having external heat exchangers. These require the pneumatic conveying of catalyst at high velocity through transfer pipes and the narrow tubes of the heat exchangers. The action of the frictional and impact forces involved on the catalyst particles, particularly so when sudden changes in direction are involved, must increase the rate of attrition. The consequent more rapid build up in the concentration of catalyst fines not only gives rise to the difficulties mentioned above but must also decrease the efficiency of the cooling system. R. M. Braca and A. A. Fried (5) have described the attrition which occurred in a fluidized-catalyst phthalic anhydride plant. No catalyst make up was used and after two years operation the proportion of catalyst particles less than 20 $\mu$  had increased from 9 to 48 per cent. This fine material plated out on the tubes of the catalyst cooler and about half of them gradually became blocked. The installation of cooling tubes within the fluidized-catalyst bed overcomes this drawback. The excellent thermal conductivity of the fluidized catalyst and the good heat-exchange characteristics of the system make this possible.
- (2) The Filters. Commercial phthalic anhydride plants differ in the disposition of the catalyst filters. Some of them house the filters in a number of separate vessels, each connected to the top of the reactor by a pipe-line. The catalyst fines which accumulate on the surface of the filters are periodically blown off and conveyed back into the base of the fluidized-bed by means of some of the fluidizing air. The attrition to the catalyst fines thereby caused will, of course, be much less than that which would obtain if coarser particles were involved. Nevertheless, it will make some contribution to the rate of catalyst attrition.

Other phthalic anhydride plants have the filter units located in the top of the reactor itself. On blow-back, the catalyst filter cake falls back into the reactor and the transfer of catalyst at high velocity through pipe-lines is thus avoided. This simple arrangement of filters, however, makes somewhat more difficult the problem of cooling the product gases issuing from the boiling catalyst bed. An increase in the height of the knock-out zone above the fluidized bed overcomes this difficulty.

- (3) The Distributor Plate. In the fluidized-catalyst reactor it is important to establish reasonably uniform distribution of air and naphthalene vapour across the whole bed, otherwise the air:naphthalene ratio would vary from place to place and hinder the attainment of optimum reaction conditions. The height:diameter ratio of the fluidized-catalyst bed and the low density of the catalyst are both favourable to good fluidization. Nevertheless, in a large fluidized reactor, say 10 ft. diameter or more, it is essential to operate with an appreciable pressure drop across the distributor plate, otherwise there is a danger of uneven air distribution. Various methods of attaining even distribution have been proposed. e.g., (a) the use of multi-jets instead of holes (9); (b) the gas is introduced through a flared, conical-shaped zone at the base of the reactor, high turbulence being maintained in this zone and a distributor plate is not used (8). Another proposal (17) is to subject the reactor, which has a conical bottom, to vertical linear oscillations, 100 per sec., with an amplitude of 1.5 mm. These methods would lead to additional catalyst attrition, particularly the one using high velocity jets. T.J.P. Pearce and I.C.I. Ltd., (10) have suggested the use of a suitable porous ceramic plate. Its maintenance in a large reactor would possibly be difficult. A method of obtaining any desired pressure drop across a distributor plate without high velocity air streams increasing catalyst attrition would be by use of the device shown in Fig. 5. Instead of a simple hole the small conical filter elements are inserted in the distributor plate. The entry holes to the filters could be made small enough to give back pressure necessary for good distribution. The air used for fluidization would have to be filtered to prevent the slow clogging of the small filters from the underside.

It is not difficult in a laboratory fluidized reactor, or in a pilot plant reactor to obtain relatively uniform fluidization and consequently a high degree of control of reaction conditions. This is evidenced by the results given in Table 2.

TABLE 2.

Results obtained in a 2 in. diam. laboratory fluidized-catalyst reactor using pure naphthalene (0.07% S).

Temp. °C	Air:Naphthalene Ratio	Yields % w/w.		
		P.A.	M.A.	N.Q.
350	15:4	104.5	1.7	0.7
330	11:5	104.1	1.1	0.9
320	14:0	104.2	1.5	1.2

Pilot plant results obtained in a 6" diam. fluidized-catalyst reactor using petroleum naphthalene (S free) c.p. 79.4°C.

Temp. °C	Air:Naphthalene Ratio	Crude Yield %	Analysis of Crude %		
			P.A.	M.A.	N.Q.
335	15:1	100.2	98.0	0.09	1.6
340	15:1	99.7	98.5	0.13	0.7
350	15:1	99.8	97.9	0.19	1.1

It is possible that the quality of fluidization and distribution in a full scale plant can be assessed by their approach to laboratory results.



### Sulphur in Naphthalene

The principal impurity in phthalic grade, coal tar naphthalene is thionaphthene which boils only  $4^{\circ}\text{C}$  above the b.p. of naphthalene. Naphthalene derived from tar from sulphurous coals may contain 1% sulphur, which is equivalent to 4.19% thionaphthene. Catalysts which have been used in the past in fluidized-bed reactors have tended to deactivate when sulphurous naphthalene is used as the feed (6). It has been stated that sulphur-free naphthalene (sodium treated) is required for optimum conversion to phthalic anhydride. This is no longer so as a sulphur-resistant fluidized-bed catalyst is now available. It has also been claimed (18) that the presence of sulphur in the naphthalene is advantageous in preventing catalyst deactivation. The oxidation of thionaphthene, however, cannot yield phthalic anhydride, so its presence in naphthalene must mean an equivalent loss of phthalic anhydride yield.

In order to determine whether or not excessive amounts of thionaphthene had any deactivating action on the new fluidized-bed catalyst the following work was carried out. Naphthalenes were prepared containing increasing proportions of thionaphthene (0 to 4.75% S) as the principal impurity. These were oxidized with air in a laboratory fluidized catalyst apparatus. The results obtained are shown in Fig. 6. There is a proportionate falling off in the phthalic anhydride yield as the thionaphthene content of the naphthalene feed increases and there is also a steady increase in the maleic anhydride yield. A final oxidation using again a sulphur-free naphthalene feed gave results quite close to those obtained initially, indicating that the excessive proportion of sulphur had not deactivated the catalyst. A sulphur free naphthalene gives a proportionately higher yield and probably also, less corrosion in the condensation system. The lower maleic anhydride content of the crude product will probably reduce distillation losses during the final purification. Whether or not pure or sulphurous naphthalene should be used as feed for a fluidized phthalic anhydride plant is a question to be answered by the economics of the operations.

Pilot plant experiments in a 6" diam. fluidized catalyst reactor were carried out using two different coke-oven naphthalenes (a) c.p.  $77.6^{\circ}\text{C}$ ; S, 0.87% and (b) c.p.  $79.6^{\circ}\text{C}$ ; S, 0.31% and Ashland petroleum naphthalene c.p.  $79.4^{\circ}\text{C}$ ; S, 0.00%. A range of operating conditions was used for each naphthalene. Table 3 gives the yields obtained under the optimum operating conditions. These results indicate that the presence of thionaphthene in the naphthalene feed does not wholly account for the reduction in yield.

TABLE 3.

<u>Naphthalene</u>	<u>Reaction Temp. <math>^{\circ}\text{C}</math></u>	<u>Average Yield of Crude %</u>	<u>Analysis of Product %</u>		
			<u>P.A.</u>	<u>M.A.</u>	<u>N.Q.</u>
Coke Oven 0.87% S	350	91.3	97.5	0.56	0.6
Coke Oven 0.31% S	340	96.1	98.5	0.18	trace
Petroleum 0.00% S	335	100.2	97.9	0.19	1.1

### Conclusion

Apart from the catalytic cracking of petroleum, the development of the fluidized-catalyst technique in the chemical manufacturing industries has been relatively slow. This has been largely due to the state of our fundamental knowledge of the properties of fluidized beds and of heterogeneous catalysis in such beds.

The methods adopted for overcoming the difficulties in developing the fluidized-bed method for naphthalene oxidation described above are already being successfully applied to other processes, particularly to exothermic oxidations. There is little doubt that the success achieved with the phthalic anhydride process will accelerate the wider industrial use of fluidized-bed catalysis.

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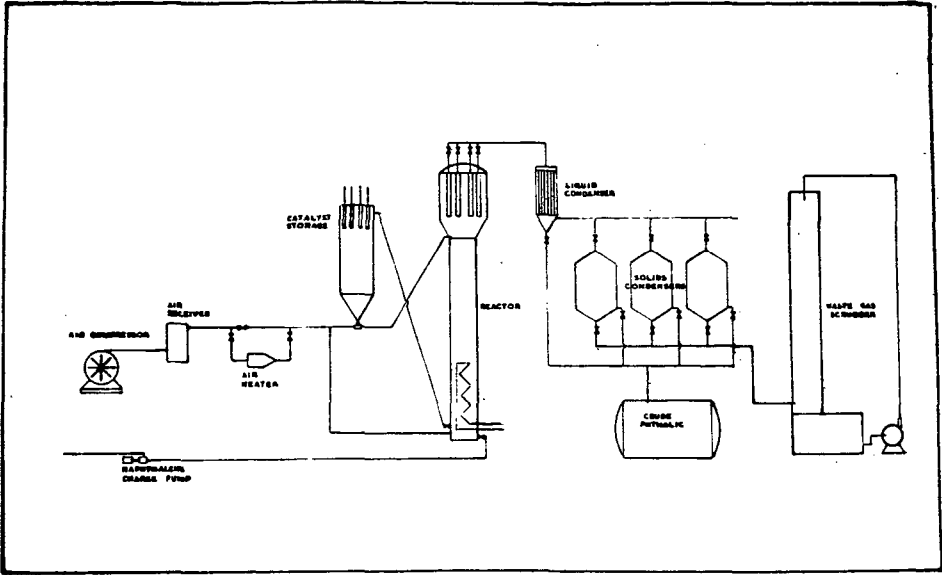


Fig. 1. Fluidized Catalyst Phthalic Anhydride Plant.

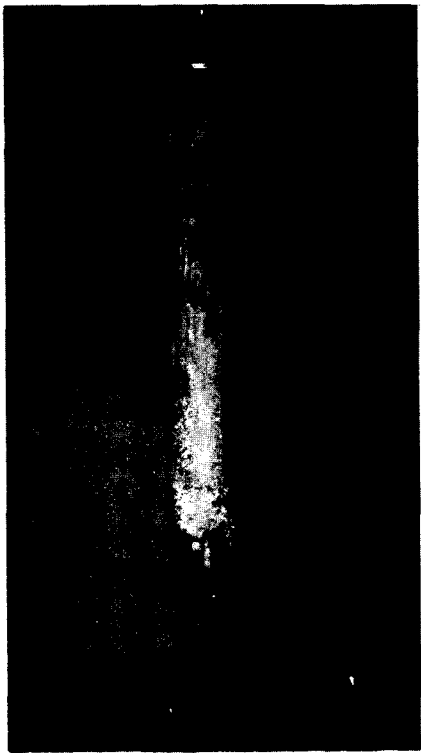


Fig. 2. Condensation of phthalic anhydride on cooled finned tube.

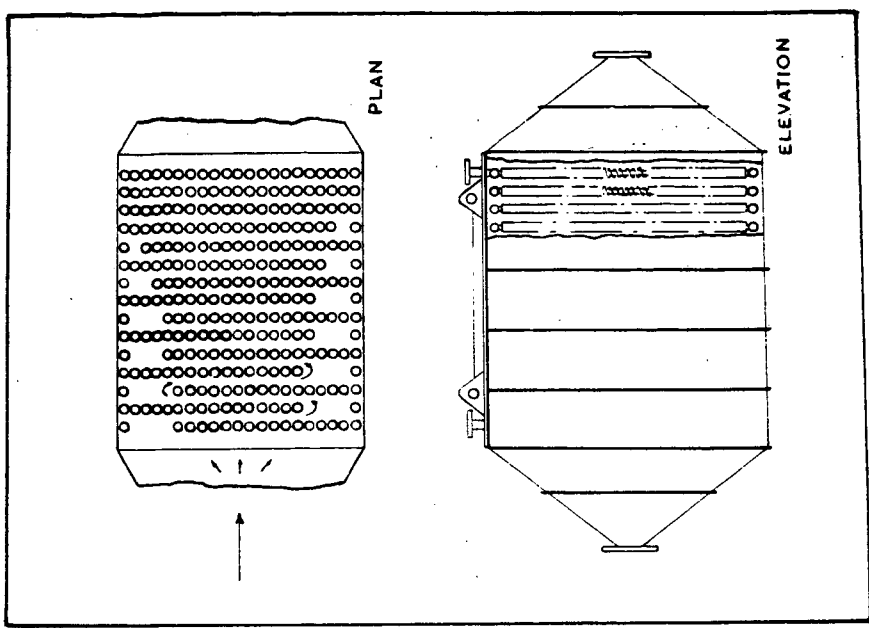


Fig. 3. Switch Condenser.

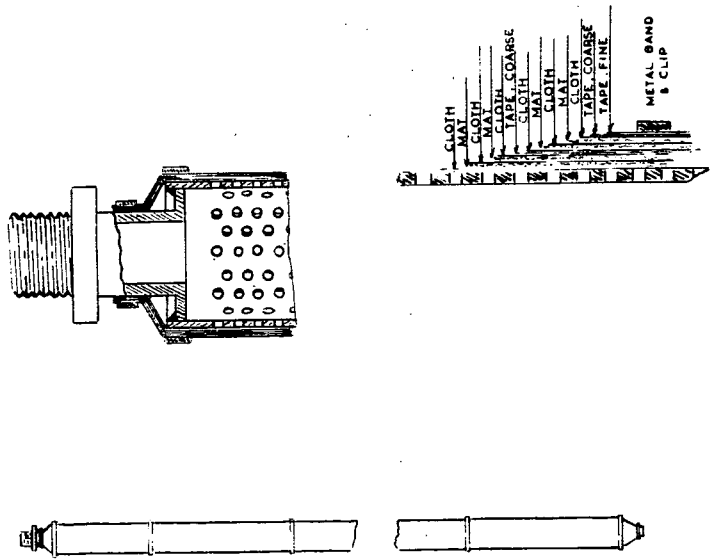


Fig. 4. Fibre-glass Filter.

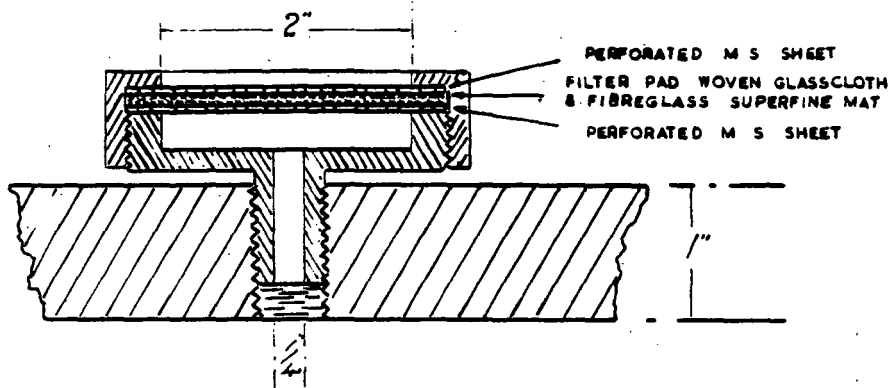


Fig. 5. Distributor Plate Filter.

